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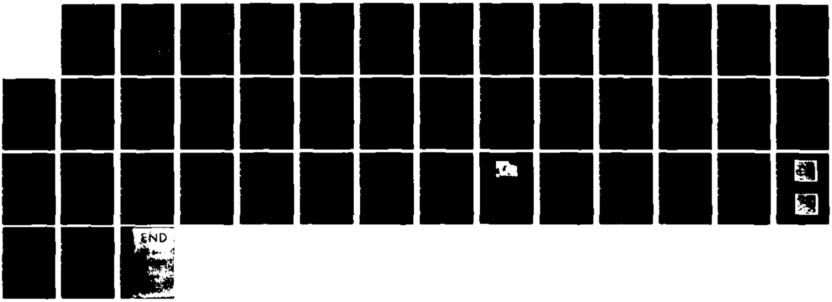

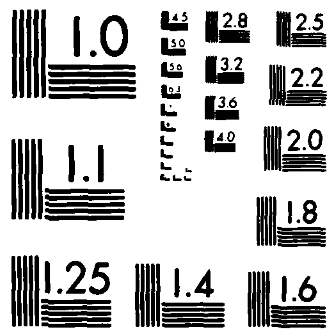

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ESR TECHNOLOGY IN AUSTRALIA - LABORATORY RESEARCH AND INDUSTRIAL DEVELOPMENT

G.M. Weston and J.C. Ritter

Approved for Public Release

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ABSTRACT

a. prov.

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Scientific and technical work undertaken jointly by MRL and the Commonwealth Steel Company, during a period of technology transfer on electroslag refining (ESR) of premium quality steels for ordnance, is reviewed in detail. Investigations used a small experimental laboratory ESR unit, and a large commercial unit manufactured by Consarc. Melting temperatures of some multi-component local slags were found to be significantly higher than reported nominal values. It was found that variations in slag composition during remelting could be assessed visually. Deoxidation practice, especially the type of deoxidant, had a greater influence than slag composition on levels of residual silicon and aluminium and on cleanliness of the final ingot steel. Silica-rich slags gave unsatisfactory ingot surfaces with higher melting point steels, while increasing slag calcium levels or introducing rare earth elements improved the modification of inclusions. Forced dry air circulation above the slag layer greatly enhanced sulphur removal from the steel. Mechanical properties, including tensile, notched impact, fracture toughness and fatigue, of as-cast ESR steel compared favourably with transverse values in forged electric furnace steel. Lower sulphur ESR steels showed improved ductility, fracture toughness and fatigue properties compared with replacement electric furnace steels, while lowering the sulphur level improved isotropy of these properties. A drop in some properties occurred at low forging reductions ($\sim$1.5 : 1), while attempts to assess ingot forgability by laboratory tests proved unsuccessful.

C O N T E N T S

ESR TECHNOLOGY IN AUSTRALIA - LABORATORY RESEARCH AND INDUSTRIAL DEVELOPMENT

1. INTRODUCTION

Australian interest in electroslag remelting (ESR) technology began in the late 1960s with information through TTCP channels that a requirement for ESR steel in defence ordnance was anticipated, a requirement which could be expected to flow on to Australia. Studies in this new technology were begun at Materials Research Laboratories (MRL) and a small experimental unit was completed in 1974 [1].

Considerable knowledge and operating experience were obtained from this unit by the time the first commercial ESR plant in Australia was commissioned in early 1979. The plant was installed by the Commonwealth Steel Company (Comsteel) at their Waratah works in Newcastle, N.S.W.

The Department of Defence maintained a close interest in establishing this plant, particularly from the strategic supply viewpoint. An informal, co-operative relationship had been built up between staff from Comsteel and MRL through the common interest in ESR. The transfer of technology achieved under this arrangement was a principal factor leading to the virtual halving of the time taken from first commissioning to successful production of low alloy steel. Premium quality ordnance steel was produced after just 9 months, so establishing an Australian capability.

The transfer of technology was formalized during 1979-80 with the part-time attachment of an MRL Research Scientist to Comsteel. This enabled the expertise and facilities of MRL to be used in solving problems experienced in the commercial plant. Throughout its history in Australia, ESR technology has remained in the hands of these two establishments.

Several programmes of research were implemented to cover major areas of technical problems. Some of these, notably those concerned with evaluating the influence of impurity levels on mechanical properties, were directed toward satisfying specific defence requirements. With the exception of

certain fatigue property studies which were cut short, all of these programmes have now been completed. Most of the effort was centred upon high-strength, low alloy steels of the types En 25 and AISI 4340 for ordnance requirements.

The aim of this review is to survey these programmes and to highlight the results of research and the solutions to production problems. A major part of the work described was planned and directed by Dr. R.C. Andrew,* previously with MRL. Much of the industrial work was undertaken by G. Ormerod of Comsteel, in co-operation with Dr. Andrew. The bulk of the work described was done during 1979 and 1980 using commercial ingots unless otherwise stated in the text.

2. SCOPE OF MRL AND COMSTEEL FACILITIES

The MRL research facility consists of a versatile experimental unit capable of producing ingots up to 250 mm diameter and 600 mm long when using the DC power mode, with electrode either positive or negative. In the more restrictive AC power mode, the unit is limited to ingots up to 150 mm diameter. Results from mechanical test on experimental, as-cast ingots were found to match closely those reported for commercial ingots of similar composition : an indication that the great difference of scale did not destroy the equality of properties. This fact confirmed the value of the MRL unit as a pilot support [2].

Facilities at MRL provide for routine fracture toughness testing and measurement of fatigue crack growth rates, while SEM and EPMA instruments are available for fracture analysis and inclusion identification.

The installation at Comsteel is a Consarc 33-tonne AC mode unit, having two outer remelting stations each with a 25,000A single phase power supply, and a larger central station coupled to both supplies. Ingots 380 mm square or 800 mm diameter are remelted in static moulds in the outer stations, while the central station provides for ingot withdrawal moulds ranging from 500 to 1100 mm diameter. Slag prefusion is usually carried out in a separate slag furance.

The Company has facilities for routine slag analysis and for inclusion assessment. The latter was originally intended for conventional ingot steel and difficulty has been experienced in rating the much finer ESR inclusions because most were below the size range covered by standards and frequently too small to be categorized optically.

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* Dr Andrew is now with Technology Transfer Council, Brisbane, Qld.

3. RESULTS OF SLAG LIQUIDUS TEMPERATURE DETERMINATION

One of the early problems facing Comsteel was that of obtaining accurate slag liquidus temperature data as such values influenced slag prefusion characteristics, as well as ingot surface finish and process economics. Initial slag melting point data was supplied for some slag systems by Consarc. However, unsatisfactory ingot surfaces were obtained with higher melting-point alloys using the recommended five component slag system. It appeared that, in this instance, the actual slag melting-point was higher than that given by the data supplied. A further difficulty was the scarcity of slag melting point data available in the open literature, with contradictory results reported by different authors for slag systems of nominally similar composition. This information could not be relied upon without verification.

It is known that chemical variations in slag components from different sources, acting in concert with slag furnace reactions, can lead to unexpected melting point variations. A programme was therefore undertaken to determine the liquidus temperature for all slag systems in use at Comsteel. These incorporated locally available slag materials. Melting point values were obtained both from laboratory fusion experiments and from slag furnace determinations. In most instances, the values were similar to the standard data available [3]. However, in three cases the new values obtained were considerably higher (Table I) than the nominal values supplied by Consarc or the open literature.

It was concluded that compositional variations in slag feed stock materials were the major factor in the higher temperatures recorded. A re-evaluation of slag liquidus temperatures was recommended whenever the source of principal slag materials was changed.

4. SURFACE CONDITION OF HIGH MELTING POINT ALLOYS

Production difficulties in obtaining a satisfactory surface finish were experienced when remelting AISI 4340 and other low alloy grades having higher melting points (Table 2). For improved steel cleanliness, the use of five component slags containing up to 12% silica were recommended by the equipment supplier, Consarc. However, severe surface rippling was found to occur when these slags were used. Depending on remelting conditions, the rippled surface could extend the entire ingot length, or at best be confined to the top end Fig. 1. In the worst cases the rippling was some 50 mm deep, and this amount of ingot surface would have to be removed prior to forging.

To overcome this problem a number of trial ingots were produced under various remelting conditions. Several ingots were produced with slags having an initial silica content which was varied between 0 and 20% (Table 3). In other instances, calcium fluoride was added continuously during remelting to maintain the slag bath fluoride content at a constant level; a fall-off of about 7-9% was previously experienced during the course of a

remelt. Both the lowering of the slag silica level and the continuous addition of calcium fluoride proved partially successful in improving ingot surface finish, but the fine degree of melt control necessary to do this was considered unattainable on a regular production basis. The use of straight aluminium [4] as the deoxidant rather than a mixed deoxidant of aluminium and calcium silicide made little difference to the extent of surface rippling.

In another approach, the slope of the melt rate ramp was increased towards the end of remelting and, although the resulting higher slag temperature did reduce the extent of top-end rippling, it also increased the likelihood of sulphur segregation. Possible segregation effects and the lack of close control over the second stage ramp limited the effectiveness of this approach. Lowering the cross fill ratio was found to markedly increase surface rippling.

Replacement of the five component silica bearing slags with a two component one of nominal composition 70% CaF_2 + 30% Al_2O_3 was finally adopted as a simple and effective measure which resulted in a significant improvement in ingot surface appearance, without undesirable side effects. This simple slag was therefore recommended for general use in remelting AISI 4340 [5].

5. EVALUATION OF SLAG COMPOSITION BY VISUAL COMPARISON

To enable rapid assessment of important slag chemistry changes during remelting, a method based on a colour comparison with standard slag samples of known composition was developed [6]. This was achieved during remelting by comparing the colour of the longitudinal fracture surface of a cooled slag cap sample with a standard set of known composition, and the closest match obtained. This method proved rapid, reasonably reliable, and cheap. It resulted in a significant reduction in the number of samples being submitted for chemical analysis.

An initial set of standard slag samples for production of deep-hardening roll steels using a nominal 49% CaF_2 + 17% CaO + 7% MgO + 17% Al_2O_3 + 10% SiO_2 slag has been completed. This method is currently being extended to cover other metal/slag combinations.

6. CONTROL OF INGOT SULPHUR LEVELS

An attempt was made to establish close and consistent control over residual sulphur levels in remelted ingots of low alloy steel by drawing upon the extensive data accumulated from experience. Both the electrode and ingot sulphur contents were recorded over an extended period of time for each alloy grade, using a wide range of slag and deoxidant combinations [7]. Typical sulphur reductions achieved for a number of these alloy systems and

slag/deoxidant combinations are shown in Table 4. Of practical significance, were the large sulphur reductions (> 70%) which were achieved even when using slags of low basicity: such marked reductions indicate that sulphur was being effectively removed from the slag during the whole remelting operation. This removal was attributed directly to the forced dry air circulating above the slag bath, a feature of the Comsteel unit, which enabled sulphur to be effectively transferred into the furnace atmosphere by the following reaction

$$(S^{2-}) + \frac{3}{2}\ \{O_2\} \rightarrow \{SO_2\} + (O_2^{\ 2-}) \qquad \text{Eq. 1}$$

where ( ) and { } denote slag and gas respectively.

Maximum sulphur reductions were only achieved when the amount of deoxidant added to the slag bath was increased above the normal recommended level. This addition was necessary to counteract the effect of the increased oxygen supply to the slag bath and so maintain its desulphurization capacity. This adjustment was to be expected from earlier work at MRL with DC remelted ingots, which had shown that slag/metal chemical reactions involving sulphur removal will only proceed if slag oxygen levels are low [8].

This work resulted in the preparation of standard tables which covered each set of remelting conditions and enabled electrode sulphur levels to be carefully selected to meet end requirements.

7. DEOXIDATION AND SLAG PRACTICE

The immediate introduction of four and five component slag systems on the advice of Consarc when remelting with static moulds presented several problems. Although these slags were claimed to give cleaner steels, they were untried in commercial units and little was known about the slags and the influence which different slag/deoxidant combinations would have on ingot cleanliness. A further complication was that these slags would be operating under a forced dry air system unique to the Comsteel installation. A study was therefore undertaken for each slag system in use, to examine the influence of different deoxidants, and deoxidant addition rates, on residual aluminium/silicon levels, steel cleanliness, and inclusion morphology. Four and five component slags as well as the more commonly used binary and ternary slag aystems were studied.

7.1 Residual Aluminium/Silicon Levels

The use of four and five component slag systems containing silicon appeared from the earlier work by Allibert et al [9] to be a practical means of controlling the final aluminium content of ingots during remelting. The following reversible exchange reaction

$$3[Si] + 2 (Al_2O_3) \rightarrow 3 (SiO_2) + 4 [Al] \qquad \text{Eq. 2}$$

where [ ] denotes slag, was shown by these workers to be operative during remelting with silica bearing slags. In line with equation 2, the use of a ternary high-alumina slag, 40% CaF_2 + 30% CaO + 30% Al_2O_3 at the Comsteel plant, increased both ingot aluminium levels and silica loss to the slag (Table 5). This trend was less marked in the five components silica bearing slag systems. These results also showed clearly that the type of deoxidant used had a far greater influence on these slag/metal exchange reactions than did the slag composition alone. The use of aluminium as a deoxidant markedly increased both ingot aluminium levels and the amount of silica transferred to the slag, while calcium silicide deoxidant significantly reduced aluminium pick-up and silica loss. A marginal increase in ingot silicon level was observed when five-component silica slags were deoxidized with excessive addition rates of calcium silicide (0.22% of melt rate).*

The influence of mixed aluminium/calcium silicide deoxidant additions was intermediate between the above single additions. The use of "Hypercal", a deoxidant containing 22.5% Al + 9.6% Ca + 1.0% Ba + 41.8% Si and 14.0% Fe also produced intermediate levels of aluminium and silicon transfer.

7.2 Steel Cleanliness

Trials were carried out in order to relate ingot cleanliness to the remelting conditions, and so determine the optimum combination for high quality steel production.

To obtain satisfactory deoxidation with a forced dry air supply above the slag bath it was found necessary to increase deoxidant addition rates above the recommended values of ~0.07 to 0.1 and 0.18% of the melt rate for aluminium and calcium silicide respectively. The inclusion ratings in the resulting ingots, averaged over a number of melts for each slag/deoxidant combination, are given in (Table 6). The values obtained here were somewhat inconclusive, as the different slag and deoxidant combinations appeared to have only a marginal influence on inclusion type and overall steel cleanliness. A higher population of aluminates was observed in circumstances where a high alumina slag and aluminium deoxidation were employed: a result which has been reported elsewhere [10]. In line with the above trend for residual aluminium/silicon levels, the present results show that ingot aluminate levels also are influenced more by the deoxidant used than slag type.

Because of the very fine nature of many of the oxide inclusions in ESR ingots (< 5 μm diameter), there is considerable doubt that the comparative

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* Per cent of melt rate is the rate of addition of a deoxidant or other substance, expressed as a percentage of the rate at which the steel is melted from the feedstock bar.

chart method used (ASTM-E45-76 Method D) was sensitive enough to detect many of the finer inclusions and, consequently, it may not have accurately revealed changes in steel cleanliness.

The introduction of three and four component slags containing Bastnasite (a naturally occurring mineral containing rare earth oxides) in combination with smaller ingot sizes (380 mm square) did considerably improve ingot cleanliness (Table 7). These slags were considered to offer the best balance of lime and alumina combined with a high desulphurising capacity. They were used successfully to remelt AISI 4340 grade material to meet the stringent supply conditions and test requirements covered by Military Specification MIL-S-8844C Class 1 (see later, Table 14).

7.3 Inclusion Morphology

The aim of this study was to determine the influence of remelting conditions, in particular slag composition, on inclusion morphology and ultimately to relate mechanical properties to inclusion characteristics. The vast majority of the globular inclusion types observed in this programme were similar to those reported when calcium and cerium modification had been carried out in general steel making practice [11,12]. Due to the very low sulphur content of the ingots examined (< 0.006 wt%) it was very difficult to locate the finely dispersed individual oxide and sulphide inclusions.

Electron probe microanalysis revealed that the very few sulphides with an elongated morphology were simple MnS particles, while the majority of fine particles were spherical in nature and had traces of calcium, magnesium, and cerium transferred from the slag. Very few individual oxide inclusions were found, most being associated with sulphur (duplex inclusions). When present alone, they usually contained the more reactive elements calcium, magnesium and aluminium. In the present work therefore, the vast majority of inclusions were of the duplex type containing traces of calcium and magnesium in both the central oxide portion and the surrounding sulphide, while cerium when present appeared to concentrate mainly in the sulphide. When using calcium silicide deoxidation the very few silicates that were observed were always associated with other oxides and sulphides.

From the low level of modifying elements present in ESR slags, significant inclusion modification could only be expected at low sulphur levels ' 0.004%), and this was supported in the present work where lenticular sulphides were still observed in steels with sulphur levels approaching down to 0.002%. As excellent transverse ductility could be expected at these sulphur levels, it was concluded that the greater uniformity and lack of directionality of mechanical properties in ESR steels results mainly from their overall cleanliness, with some inclusion modification being an added advantage.

8. HOT WORKABILITY OF ESR AND ELECTRIC FURNACE STEEL

To obtain a quantitative comparison between the hot workability of ESR and electric furnace steels, and to test forge shop observations that ESR ingots can be deformed more and at a faster rate than electric furnace ingots during each forging cycle, two different test programmes were undertaken.

In the first series of tests, small cylinders from five steel grades were upset at high temperature and hot workability measured in terms of (a), the final cylinder height at the maximum press loading, and (b), the height at which surface rupture was first observed (Table 8). These results show little difference in the behaviour of steels from the two processing routes. The height of several cylinders for each steel type was continuously monitored throughout the upsetting operation. Typical curves for two steel grades (Fig. 2) show that the final cylinder height is less for the lower alloy grades such as AISI P20, and that deformation commences at a lower load for these steels and continues through to the limit of the press capacity.

In the second series of test, hot impact tensile test pieces similar to those used in earlier work [13] were fractured under argon at several temperatures within the range 1050-1255°C (Fig. 3). Here again, little difference was observed in the hot workability of the as-cast material from both processing routes and while the forged ESR steel did show a higher hot ductility than the above material, comparative results for a similar forged electric arc steel were not available.

As both the above test procedures failed to substantiate forge shop experience and find any significant differences in hot workability between ESR and EF samples, the validity of using small test specimens to represent the forging behaviour of large ingots is open to question. This is most likely the result of the small material volumes involved and the absence of ingot surfaces known to influence forgability.

9. PROPERTY EVALUATION OF ORDNANCE STEELS

9.1 As-Cast Properties : Tensile Strength and Notch Toughness

Although the major Australian demand is at present for premium quality ESR forging ingots, the limited range of ingot size commercially available has initiated interest in the use of ESR steel products in the as-cast and heat treated condition, as well as in products where only small forging reductions, of the order of 1.5:1, may be possible. Previous investigations [14,15,16] have shown that the general properties of as-cast ESR steel approach those obtained in the as-forged condition. Of particular interest in the field of gun barrel manufacture, Underwood [17] showed that fatigue crack growth rates and fracture toughness values were similar for the same ESR ordnance steel in both the as-cast and forged conditions. From these results it appears that the as-cast material may provide a shorter and more

economical processing than forging for some defence applications. Moreover, the evaluation of as-cast properties was considered to provide an excellent guide to ingot integrity and hence remelting practice at Comsteel.

A general comparison of mechanical properties of as-cast ingots from the MRL ESR unit, Comsteel and overseas sources is given in Tables 9 and 10. Properties of the equivalent wrought steel are included where possible. The as-cast properties of the Comsteel ingot remelted using a silica bearing slag and calcium silicide deoxidation compared very favourably with similar as-cast material supplied by an overseas manufacturer for defence evaluation. After taking into account the slightly higher strength level of the overseas material, which takes some toll of toughness and ductility, the considerably higher notch toughness and the slightly higher ductility (reduction-in-area and elongation) of the Comsteel ingot are judged to result from the greater overall cleanliness of this material.

Examination of fracture surfaces of the overseas material revealed large elongated sulphide inclusions, located at grain boundaries and oriented in the ingot growth direction. Similar inclusions have been observed in ingots with high oxygen levels produced at MRL [2], suggesting that deoxidation of the overseas material was inadequate. By contrast, the very fine dispersion of both sulphides and duplex inclusions in the Comsteel material indicated excellent remelting control.

The properties of the as-cast Comsteel material exhibited greater isotropy than after a forging reduction of 5:1, and compared favourably with the transverse wrought properties. Greater isotropy in as-cast mechanical properties is to be expected because the elongation of sulphide inclusions during hot working is known to be a major factor in lowering transverse properties, particularly ductility [18]. It has been reported [10] that the properties of as-cast material can be less isotropic than the forged material even after reductions of up to 8:1, in very large ingots. The less favourable heat transfer characteristics associated with such large ingots, especially towards the top end, appear to increase local solidification times resulting in a concomitant coarsing of the solidification structure. Experience at MRL [2] has shown that unfavourable grain boundary orientation within the test piece and inadequate deoxidation practice leading to large grain boundary sulphides can also lower as-cast transverse toughness.

It is apparent from Tables 9 and 10 that the forged Comsteel material again compared favourably with similar overseas steel supplied for gun barrel manufacture. The initial ingots supplied by Comsteel for ordnance manufacture comfortably exceeded the specified transverse mechanical properties and in general were free from the larger Type A inclusions (elongated sulphides). These inclusions are usually present in electric furnace steels even when triple slagging techniques are employed.

9.2 As-Cast Properties : Fracture Toughness and Fatigue Resistance

An evaluation of the as-cast fatigue and fracture toughness properties has been completed only for laboratory size ESR ingots and a

comparable wrought electric furnace steel. Preliminary results were nevertheless encouraging, with the fatigue crack growth values for both testing directions being similar in as-cast ESR steel. Longitudinal and transverse properties are compared in Fig. 4. Whilst the longitudinal fatigue resistance of the as-cast ESR cannot match that of the wrought electric furnace material, the transverse ESR steel is somewhat surprisingly better and fully matches the wrought EF sample. Overall, however, the spread in curves is small and individual differences are not highly significant.

Fracture toughness of as-cast material tended to be more isotropic and approached that obtained for the transverse wrought condition (Table 11). As the sulphur level of 0.020% for the above steels is much higher than the usually accepted level of 0.005% - 0.010% for high quality ordnance steels, the above results should be viewed with caution because the high sulphur level should have an exaggerated influence on wrought transverse toughness. A further advantage of the as-cast material arises from the reduced local solidification times experienced by smaller ingots. This results in a finer inclusion population.

9.3 Influence of Deformation Ratio on Mechanical Properties

To examine the change in mechanical properties as a function of deformation ratio, the as-cast Comsteel ingot I (Table 9) was forged to a number of reduction ratios between 1:1 and 28:1. The resultant property changes with forging are shown in Fig 5, and the variation in anisotropy ratio of these properties (longitudinal value/transverse value) as a function of deformation ratio (initial ingot diameter/final diameter) is shown in Fig. 6. Although most properties increased as a result of hot working, a localized dip in the yield and tensile strength occurred at a forging ratio of about 1.5:1, largely as expected from previous observations [20]. Although reasons for this dip are unclear, it has been suggested that small forging reductions open up voids around inclusions; larger forging reductions have the effect of healing these voids and restoring strength. Further study of this phenomenon is the basis of a current defence R&D contract with the Commonwealth Steel Company.

Whilst the anisotropy ratio for both the YS and UTS remained almost constant with increasing reductions, its value for the other properties increased steadily (Fig. 6). As sulphur levels of < 0.004% have been previously suggested [18] as sufficient for the elimination of the anisotropy of mechanical properties, the level of sulphur in ingot I here of 0.008% is still comparatively high. On this matter, elongated sulphide particles were observed in other aspects of the present work in steel with a sulphur level as low as 0.002%, suggesting that the value of 0.004% still lay above the threshold level for the disappearance of anistropy arising from elongated sulphide inclusions.

9.4 Influence of Sulphur on Tensile, Notch Toughness, Fracture Toughness and Fatigue Properties of Wrought Steels

As the initial defence orders placed with Comsteel covered a range of sulphur levels, sufficient data were at hand to permit an evaluation of the influence of this element on critical mechanical properties such as 0.2% proof stress, UTS, ductility, notch toughness, fracture toughness and fatigue crack growth rates for AISI 4340 and En 25 grades of steel. Strength levels were similar in all cases. The chemical compositions, tensile properties, notch toughness and cleanliness ratings (using ASTM - E45 - 76 method D) are given in Tables 12 and 13, along with data for a comparative electric furnace steel. Besides sulphur removal, the other important compositional change to occur during remelting of these steels was the lowering of oxygen from ~ 50 ppm in the electrode material to ~ 25 ppm in the remelted ingot.

The higher sulphur En 25 types (~ 0.025%) in the above table were typical of those steels supplied for small arms manufacture where there was a stringent requirement for a low level of Type B (aluminate) inclusions along with a relatively high sulphur content to assist machinability. A remelting practice involving five component silica-bearing slags and calcium silicide deoxidation, combined with resulphurization during remelting, was developed to satisfy these requirements. The intermediate sulphur level of 0.005% is typical of steels supplied for gun barrel manufacture, while the 4340 grade (0.002%S) steel was produced to satisfy the most stringent requirements of overseas orders.

It is evident from these results that, at higher sulphur levels, there is no mechanical property advantage to be gained by using ESR steel in preference to electric furnace steel. In the present ordnance application, the lower population of aluminates improved machine tool life above that experienced previously with the electric furnace steel. A further comparison of the properties in Table 12 shows that the transverse ductility and notch toughness values for the lower sulphur ESR steels were as high as 70 - 80% of the longitudinal figures, compared with only 40 - 50% for the higher sulphur ESR and electric furnace steel. The anisotropy ratio for the above properties is therefore closely related to sulphur content.

The influence of sulphur content on fracture toughness and fatigue crack growth rates is shown in Table 14 and Fig. 7. These results demonstrate that both the high sulphur ESR and electric furnace steels perform poorly in comparison with the lower sulphur ESR steels. Fracture toughness data suggests that lower sulphur ESR steels are a good 50 to 60% tougher in terms of stress intensity factor compared with the higher sulphur steels. Other factors being equal, this means that the cleaner ESR steels can tolerate a crack of at least twice the size before final fracture occurs.

Fatigue crack growth curves, generated from an almost continuous computer printout of data and summarized by Fig. 7, again emphasize the advantages to be gained at lower sulphur levels. The slope of the linear regime of the curves decreases with decreasing sulphur levels. The beginning of the upturn of these curves at high levels of alternating stress intensity occurs at $\Delta K \approx 90$ in the low sulphur steels compared with $\Delta K \approx 50$ in the high

sulphur material. This upturn signals the onset of significant amounts of static overload fracture, and marks the point at which safe operation of a component can no longer be guaranteed. It has been claimed [21] that these two differences in the fatigue crack growth curves represents a large advantage of between 2 and 3 times in the fatigue life of components such as gun barrels.

Examination of the fatigue fracture surfaces (Fig. 8) shows the larger inclusion types typical of both electric furnace and ESR steel at higher sulphur levels compared with the largest inclusion observed at lower sulphur (0.006%) levels. Furthermore, at higher sulphur levels, slag systems would be expected to have little influence on inclusion morphology.

The necessity for very clean steel became evident during development of a remelting practice to meet overseas contracts to the Military Specifications MIL-S-8846 - Class I (see Table 15). This specification required 4340 grade material to have an average transverse reduction of area of 30% at levels of 0.2% PS approaching 1500 MPa. To meet these requirements several developmental ingots were made using three and five-component slag systems; however the transverse reduction in area properties could only be achieved when remelting was carried out using bastnasite-containing slags, in conjunction with a smaller ingot size (380 mm square) to reduce the local solidification time. These slags were considered to offer the optimum combination of lime and alumina levels with a high desulphurising capacity which reduced ingot sulphur levels to 0.002%. These remelting conditions produced an extremely fine dispersion of inclusions, mainly duplex. A typical inclusion rating for this steel is given for ingot ESR 4340-2 Table 13.

It can be concluded that although inclusion modification may improve transverse properties at low sulphur levels, it is the overall improvement in steel cleanliness, especially the population of sulphide inclusions; which is the dominant factor in the production of high quality ordnance steels.

10. CONCLUSIONS

In all, the co-operative effort of research and development has been considered most valuable to both MRL and industry, and highly successful in achieving its goal of establishing an Australian capability for producing low alloy ESR steels of premium quality for ordnance and other defence needs.

The following summary points are drawn from the work:

1. Slags high in silica caused surface finish difficulties when remelting certain low alloy steels.

2. Large sulphur reductions were obtained in the refining unit at Comsteel, a result of using forced dry air circulation above the slag bath.

3. The choice of deoxidant was found to have more influence than the slag chemistry on the inclusion content and residual levels of Al and Si in the final ingot.

4. The general mechanical properties of as-cast ESR steels were found to approach those for wrought electric furnace steels.

5. Low-sulphur ESR steels had better fatigue and fracture toughness properties than electric furnace steels.

6. Contrary to forge shop comments, laboratory tests showed that ESR steel did not have superior hot workability over electric furnace steels.

7. The anisotropy ratio for Charpy-V impact toughness and reduction in area, increased with sulphur content.

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TABLE 1

LIQUIDUS TEMPERATURES OF COMMERCIAL ESR SLAGS

Sample Type+	Slag Composition, Wt%							Liquidus Temperature °C		
	CaF_2	CaO	MgO	Al_2O_3	SiO_2	Bastnasite	Total	MRL	Consarc++	Barraclough
Nom.	70(70)	-	-	30(30)	-	-	100			1320-1340
S.F.	63.3	5.9	0.44	28.2	0.9	-	98.7			
MRL	61.3	6.5	0.1	27.5	0.6	-	95.9	1450-1480		
Nom.	60	20	-	20	-	-	100			1240-1260
MRL	54.5	18.6	0.1	23.0	0.7	-	96.7	1390-1405		
Nom.	65(70)	15(10)	-	10	10	-	100		1265	
S.F.	54.6	22.4	0.8	10.0	9.4	-	97.2			
MRL	57.8	18.5	0.1	10.4	10.5	-	97.3	1365		
S.F.	63.7	18.2	0.4	11.1	7.0	-	100.4			
MRL	61.7	13.8	0.1	13.3	9.1	-	98.0	1355		
S.F.	55.8	13.4	3.0	18.6	6.0	-	96.8			
MRL	55.2	14.9	0.1	14.9	12.7	-	97.8	1355-1370		
S.C.	53.3	19.5	0.3	14.0	9.7	-	96.8			
MRL	54.8	16.4	0.1	15.5	13.4	-	100.2	1355-1370		

++ Supplied by Consarc Corporation
K.C. Barraclough, "Refining by Remelting", Metals Society Conference "Sheffield Steelmaking - A Decade of Progress", Sheffield, July, 1975.

\+ Nom. - nominal composition. Actual composition charged to the slag furnace is shown in parenthesis
S.F. - slag bath sample at the start of the remelting operation
S.C. - slag bath sample at the end of the remelting operation
MRL - Laboratory sample

TABLE 2

COMPOSITIONS AND MELTING POINTS OF LOW ALLOY STEELS STUDIED

Grade	Composition (wt%) C	Si	Mn	P	S	Ni	Cr	Mo	Nominal Melting Point (^{o}C)
AISI P20	0.32	0.60	0.75	0.030	0.030	0.10	1.70	0.40	1506
En 24 and AISI 4340	0.40	0.30	0.70	0.030	0.030	1.50	1.00	0.30	1495
† 3% Cr Roll Steel (DHC)	0.90	0.60	0.30	0.030	0.030	0.10	3.0	0.30	1463

† A Comsteel Deep Hardening Chromium Roll Steel

TABLE 3

TYPICAL COMPOSITION RANGE FOR FIVE COMPONENT SLAGS

USED TO REMELT LOW ALLOY STEELS

Position	Slag Composition (wt%)							
	CaF_2	CaO	MgO	Al_2O_3	SiO_2	FeO	Mn	Total
S.F.	38.8	20.8	6.4	12.9	20.4			99.3
S.C.	33.2	21.7	6.0	15.2	22.4			98.5
S.F.	45.2	11.9	10.2	18.0	10.35			95.65
S.C.	38.2	14.5	6.5	17.4	16.03			92.63
S.F.	62.4	16.6	0.13	17.95	0.19	1.37		98.64
S.C.	55.1	14.7	0.20	16.90	6.56	0.25		93.71
S.F.	45.6	11.5	7.38	15.4	9.05	0.39	<0.01	89.33
S.C.	41.2	12.4	6.26	19.0	12.83	0.77	0.60	93.06
S.F.	43.7	16.1	6.52	18.0	4.82			89.14
S.C.	50.5	14.3	5.90	15.8	4.27			90.77
S.F.	43.0	12.1	10.6	22.0	1.35			89.05
S.C.	39.8	17.5	5.93	20.2	11.07			94.5
S.F.	46.6	16.9	7.5	21.2	5.50	0.13	<0.01	97.84
S.C.	43.1	16.1	5.2	21.9	11.40	0.98	0.46	99.14
S.F.	55.68	13.0	6.47	10.70	4.41	0.08	<0.01	90.35
S.C.	47.01	14.9	6.47	19.35	10.09	0.36	0.40	98.58
S.F.	51.99	13.6	6.47	16.3	6.59	0.03	<0.01	94.99
S.C.	44.93	14.6	5.47	16.5	12.42	0.62	0.76	95.3
S.F.	57.51	11.1	8.29	17.1	3.69	0.10	<0.01	97.8
S.C.	50.10	14.9	5.47	17.0	9.44	0.33	0.46	97.7
S.F.	49.32	15.0	6.8	20.8	7.1	0.06	<0.01	99.08
S.C.	-	-	-	-	11.0	0.64	0.40	-

S.F. Slag furnace sample

S.C. Slag cap sample

TABLE 4

LEVELS OF DESULPHURIZATION ACHIEVED FOR VARIOUS SLAG/ALLOY COMBINATIONS

Alloy/Slag Combination	Deoxidant (Nominal Rate)	Electrode Sulphur Content (wt%)			
		0.010	0.010-0.019	0.020-0.030	0.030
AISI P20 70F/0/0/30/10	Al(0.08)			74% (2)*	
AISI 4340/ 65F/15/0/0/0 + 25% Bast.	Al(0.10)		88% (1)		
AISI 4340/ 62F/10/0/17/0 + 11% Bast.	CaSi(0.18)				
AISI 4340/ 70F/15/0/15/0	CaSi(0.18)		88% (4)		
† 3% Cr Roll Steel 65F/15/0/10/10	CaSi(0.10)				45% (1)
AISI 4340/ 65F/15/0/10/10	Al(0.10)		69% (2)		
+ En 24 etc.	Al(0.05)			77.4% (3)	
40F/30/0/30/0	Al(0.08)			69.7% (6)	
3% Cr Roll Steel	Al(0.05)	83% (8)	87% (10)	89% (4)	
40F/30/0/30/0	Al(0.07)		72% (2)		86% (2)
"	Al(0.08)			82% (1)	
"	Al(0.10)		85% (1)		
"	CaSi(0.13)		77% (2)		
"	CaSi(0.18)			91% (2)	

* The number of ingots for which Data was available is shown in parenthesis.

+ En24, En25, 4130, 4140, 4340.

† Comsteel
Deep Hardening Chromium Roll Steel

TABLE 5

SILICON AND ALUMINIUM VARIATION WITH DIFFERENT SLAG/DEOXIDANT COMBINATIONS

SLAG TYPE	Deoxidant Type	Deoxidant Rate	No Melts	Steel Type	Al Electrode	Al Ingot	Al Change	Si Electrode	Si Ingot	Si Change
49/17/7/17/10	CaSi	0.1	5	AISI-H13	0.002	0.0045	-0.0025	1.08	1.05	-0.03
" "	Al	0.1	3	" "	0.006	0.028	+0.022	1.18	1.0-1.15	-(0.03-0.18)
" "	Al + CaSi	0.06 0.08	4	" "	0.004	0.021	+0.017	1.03	1.0	-0.03
" "	CaSi	0.18	2	AISI-4340 En-25	0.003	0.005	+0.002	0.65	0.65	NIL
" "	Al + CaSi	0.05 0.08	2	" "	0.021	0.031	+0.01	0.29	0.26	-0.03
" "	Al + CaSi	0.06 0.08	4	" "	0.002	0.02	+0.018	0.25	0.25	0
40/30/0/30	Al	0.1	3	" "	0.003	0.031	+0.028	0.59	0.52	-0.07
" "	CaSi	0.22	4	" "	0.004	0.14	+0.01	0.57	0.61	+0.04
" "	Al + CaSi	0.05 0.08	4	" "	0.005	0.018	+0.013	0.59	0.56	-0.03
" "	Hypercal	0.18	3	" "	0.002	0.016	+0.014	0.61	0.58	-0.03

TABLE 6

MICRO-INCLUSION LEVELS FOR A NUMBER OF SLAG/DEOXIDANT COMBINATIONS

Melt Type	Slag* Type	Deoxidant used	Average Worst Field Cleanliness Rating			
			A	B	C	D
En25	70/0/0/30	Al	1.00H	0.35T	-	1.05T/H
"	40/30/0/30/0	Al	0.60H	0.30T	-	0.80T/H
"	"	CaSi	0.15H	0.10T/H	-	1.50T
"	"	Hypercal	0.50H	-	-	0.90T/H
AISI 4340	70/15/0/10/10	Al	0.20H	0.30T	-	1.20T
AISI 4340	65/15/0/10/10	Al	0.75H/T	0.35T	-	0.55T
En25	65/15/0/10/10	CaSi	0.90H	-	-	0.85T
"	49/17/7/17/10	Al	0.65H	0.25H	-	1.10T
"	"	CaSi	0.60H	0.1T	-	0.75T
"	"	Al/CaSi	0.60H	0.20H	-	0.70T
"	"	Hypercal	0.50H	0.15T	-	1.00T

* Slag constituent order CaF_2 + CaO + MgO + Al_2O_3 + SiO_2

TABLE 7

MICRO-INCLUSION LEVELS IN 4340 STEEL REMELTED

TO MEET MILITARY SPECIFICATION MIL-S-8844C CLASS 1

Slag Type	Deoxidant	Worst Field A	B	C	D	Rateable Fields A+B+C	D
+ 65/15/0/10/10	Al	1.0H	1.0T	-	1.0T	7	4
65/10/0/10/10	Al	1.0T	-	-	-	3	NIL
65/15/0/0/0 + 25 Bas*	Al	-	-	-	1.0T	NIL	4
65/15/0/17/0 + 11 Bas	CaSi	-	-	-	1.5T	NIL	4
65/15/0/10/10	Al	1.0H	1.0T	-	1.0T	5	1

+ Slag constituent order CaF_2 + CaO + MgO + Al_2O_3 + SiO_2.

* Bastnasite - A naturally-occurring element containing rare earth oxides.

TABLE 8

COMPARISON OF THE PERFORMANCE OF CYLINDERS FROM ESR AND ELECTRIC FURNACE INGOTS AFTER UPSETTING

Grade	Job Temp. °C	Cylinder Location Height mm	ESR INGOT Mean Rupture Height mm	ESR INGOT Mean Final Height mm	ELECTRIC FURNACE INGOT Mean Rupture Height mm	ELECTRIC FURNACE INGOT Mean Final Height mm
AISI-D2	1110	Surface	70 (5 of 5) Scale 5	56.8	55 (3 of 3) Scale 5	52.3
		Centre	65 (5 of 5) Scale 5	54.6	65 (3 of 3) Scale 5	54.5
† 3% Cr Roll Steel	1180	Surface	Nil	38.0	Nil	37.8
		Centre	45 (2 OF 3) Scale 2	39.3	45 (3 OF 3) Scale 2	39.7
AISI-H11	1180	Surface	Nil	46.4	Nil	46.7
		Centre	50 (2 of 5) Scale 2	46.7	50 (1 of 3) Scale 2	44.0
AISI-P20	1200	Surface	Nil	37.3	Nil	38.7
		Centre	50 (2 of 3) Scale 1	36.8	40 (1 of 3) Scale 1	38.7
En26	1200	Surface	Nil	37.0	Nil	39.3
		Centre	Nil	36.8	Nil	39.2

The job temperatures may appear low but they are representative of forge plant practice.
The degree of rupture is given in terms of a relative scale ranging from 1 (minor) to 5 (severe).
The number of cylinders ruptured is shown in parenthesis.

† Comsteel Deep Hardening Chromium Roll Steel

TABLE 9

COMPARISON OF TENSILE PROPERTIES FOR ESR GUN BARREL STEELS

Material Identification	Sulphur Content wt%	Longitudinal Results: 0.2% Proof Stress MPa	Longitudinal Results: Tensile Strength MPa	Longitudinal Results: % Elong	Longitudinal Results: Reduction in Area %	Longitudinal Results: Fracture Strength MPa	Transverse Results: 0.2% Proof Stress MPa	Transverse Results: Tensile Strength MPa	Transverse Results: % Elong	Transverse Results: Reduction in Area %	Transverse Results: Fracture Strength MPa
Lab. ESR En 25 As-Cast	0.019	1090 1090	1170 1170	13 14	44 50	1660 1760	1090 1090	1170 1180	11 9	37 23	1580 1380
Lab. ESR En 25 As-Cast	0.010	1090 1100	1190 1190	13 12	48 46	1760 1700	1070 1080	1160 1170	13 13	41 43	1620 1640
Overseas ESR En 25 As-Cast	0.004	1130	1260	8	22	-	1120	1220	9	29	-
Comsteel ESR Ingot I As-Cast	0.008	1070	1120	11.5	40.4	-	1066	1120	10	35	-
Comsteel ESR Ingot I Forged	0.008	1090	1130	14	60.1	-	1080	1130	13	47	-
Overseas ESR	0.005	-	-	-	-	-	1080	1160	15	48	-
En 25 Forged	0.011	-	-	-	-	-	1240	1330	13	46	-

TABLE 10

RESULTS OF CHARPY IMPACT TESTS FOR ESR GUN BARREL STEELS

Material Identification	Nominal Diameter, mm	Sulphur Content, wt%	Notch Impact Toughness at ^{o}C, Joules																
			Longitudinal Results									Transverse Results						
			-196	-150	-120	-80	-40	-30	0	20	80	-196	-120	-80	-40	-30	20	80
Lab. ESR En 25 As-Cast	100	0.019	7		15	25 25	26	26		31	34	4	15	18	18	19	25	30
Lab. ESR En 25 As-Cast	100	0.010	5 8	15	18	22	35	37		42	44	5	15	16	24	27	29	31
Overseas ESR En 25 As-Cast	800	0.004					20			40					15		28	
Comsteel ESR Ingot I As-Cast	800	0.008					57								48			
Comsteel ESR Ingot I Forged		0.008					80								55	68		
Overseas En 25 Forged		0.005 0.011													54 28			

TABLE 11

FRACTURE-TOUGHNESS TEST RESULTS FOR EXPERIMENTAL

AS-CAST ESR AND WROUGHT ELECTRIC FURNACE STEEL

Sample	Sulphur Content wt%	Test Direction	K_Q MPa $m^{1/2}$	Overall Validity	K_{Ic} MPa $m^{1/2}$	K_J MPa $m^{1/2}$
As-Cast En-25	0.02	Trans.	101	yes	101	
		Long.	109	yes	109	
		Long.	116	no		122
Wrought En-25	0.02	Trans.	107	yes	107	
		Long.	154	no		197
		Long.	154	no		182

Specimens were standard 25.4 mm thick CTS in all cases.
0.2% proof stress values from Table 3 used in validity calculations.
All tests performed at room temperature (+21°C).
K_{Ic} is plane strain fracture toughness as defined by ASTM E 399.
K_Q is the provisional value of K obtained by test, without being subject to validity criteria.
K_J is the value of fracture toughness obtained from a J-integral test and expressed in units of (MPa $m^{1/2}$).

TABLE 12

COMPOSITION AND MECHANICAL PROPERTIES OF ESR AND ELECTRIC FURNACE LOW ALLOY STEELS

GRADE	CHEMICAL COMPOSITION								MECHANICAL PROPERTIES						
	C	Si	Mn	P	S	Ni	Cr	Mo	TEST DIRECTION	0.2% PS N/mm^2	UTS N/mm^2	EI %	RA %	CVN IMPACT $-40^{o}C$, J	ANISOTROPY RATIO T/L RA
F 4140	.45	.23	.84	.026	.036	.16	.92	.17	LONG	900	990	16	59.0	83	
									TRANS.	895	990	6	22.0	28	0.37
SR 4340-1	.42	.26	.79	.010	.006	1.86	.82	.25	LONG	1170	1255	12	52.0	60	
									TRANS.	1145	1235	9	35.0	42	0.67
SR 4340-2	.41	.22	.73	.007	.002	1.78	.79	.23	LONG	1150	1235	13	53.0	66	
									TRANS.	1160	1230	11	41.0	56	0.77
F En25	.30	.26	.67	.021	.025	2.58	.65	.50	LONG	1050	1150	12	54.0	75	
									TRANS.	1020	1135	9	26.0	40	0.48
SR En25-3	.32	.28	.64	.016	.03	2.38	.68	.52	LONG	1070	1123	13	58.0	74	
									TRANS.	1062	1110	9	24	38	0.48
SR En25-1	.33	.28	.65	.020	.006	2.40	.61	.46	LONG	1110	1205	13	59.0	61	
									TRANS.	1095	1205	11	49.0	48	0.83
SR En25-2	.31	.29	.63	.014	.006	2.38	.91	.60	LONG	1075	1165	14	62.7	93	
									TRANS.	1080	1165	13	50.0	70	0.80

E.F. - Electric arc furnace vacuum degassed.

TABLE 13

CLEANLINESS RATINGS FOR A RANGE OF DEFENCE STEELS HAVING DIFFERENT SULPHUR LEVELS

STEEL GRADE	CLEANLINESS (ASTM E45-METHOD D-WORST FIELD RATING)			
	A	B	C	D
+EF AISI 4140	2.00H	0.30HT	0	0.70T
ESR AISI 4340-1	0.60H	0	0	1.00T
ESR AISI 4340-2	-	-	-	1.00T
EF En-25	1.7H	1.20T	0	0.70T
ESR En-25-1	0.50H	0.15T	0	1.00T
ESR En-25-2	0.50H	0	0	1.00T
ESR En-25-3	1.4H	0	0	1.00T

\+ EF - Electric furnace vacuum degassed.

TABLE 14

COMPARISON OF TRANSVERSE FRACTURE TOUGHNESS VALUES FOR DIFFERENT SULPHUR LEVELS

STEEL	J_{Ic}, kN/m	$^{*}K_J$, MPa $m^{1/2}$	Fatigue Constants† C	m
EF-En25	61	118	3.72×10^{-26}	2.57
ESR En25-3	45	92	2.85×10^{-27}	2.65
ESR AISI 4340-1	147	183	2.82×10^{-33}	2.10
ESR AISI 4340-2	$^{**}K_{EE}$ = 185			

J_{Ic} Is J-integral fracture toughness as defined by ASTM E813-81.

* Fracture toughness derived from the J_{Ic} value

† From the Paris-Erdogan law relationship between fatigue crack growth rate and stress intensity range, $da/dN = C\Delta K^m$.

** Estimation of K_c based on equivalent energy method of Witt [22], and promulgated as ASTM draft proposal.

TABLE 15

TRANSVERSE TENSILE PROPERTIES

Material	U.T.S. (MPa)	0.2% P.S. (MPa)	Elongation (%)	R of A (%)
MIL-S-8844C				
Class I	1792 Min	1496 Min	6 Min	25 Min., 30 Av.
AISI 4340	1865	1643	8	28.0 *
	1803	1603	10	27.8 *
	1800	1578	8	29.4 +
"	1824	1578	7	17.2
	1807	1540	8	22.6
"	1824	1563	10	34.4 †
	1824	1578	10	34.4 †
"	1839	1563	10	36.0 †
	1848	1563	10	36.0 †
"	1857	1563	8	26.3
	1853	1578	8	28.0

* Ex 150 x 150 mm bloom

\+ Ex 300 x 300 mm bloom

† Remelted with slags containing Bastnasite.

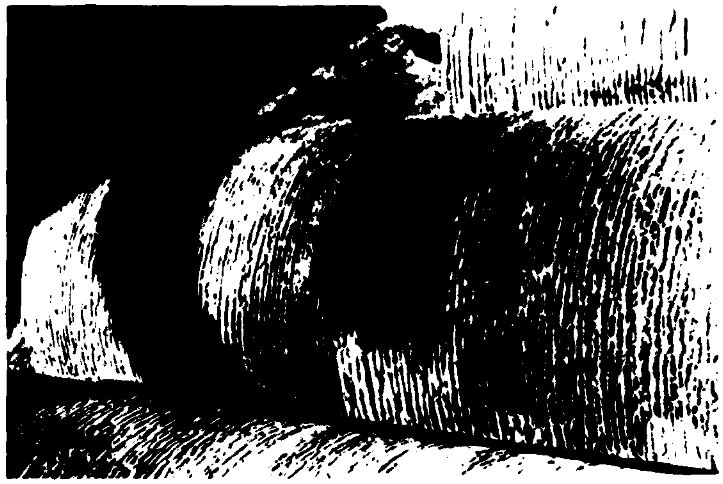


FIG. 1 Rippled surface of ingots of ESR low alloy steel, AISI 4340 remelted using high-silica, five-component slags.

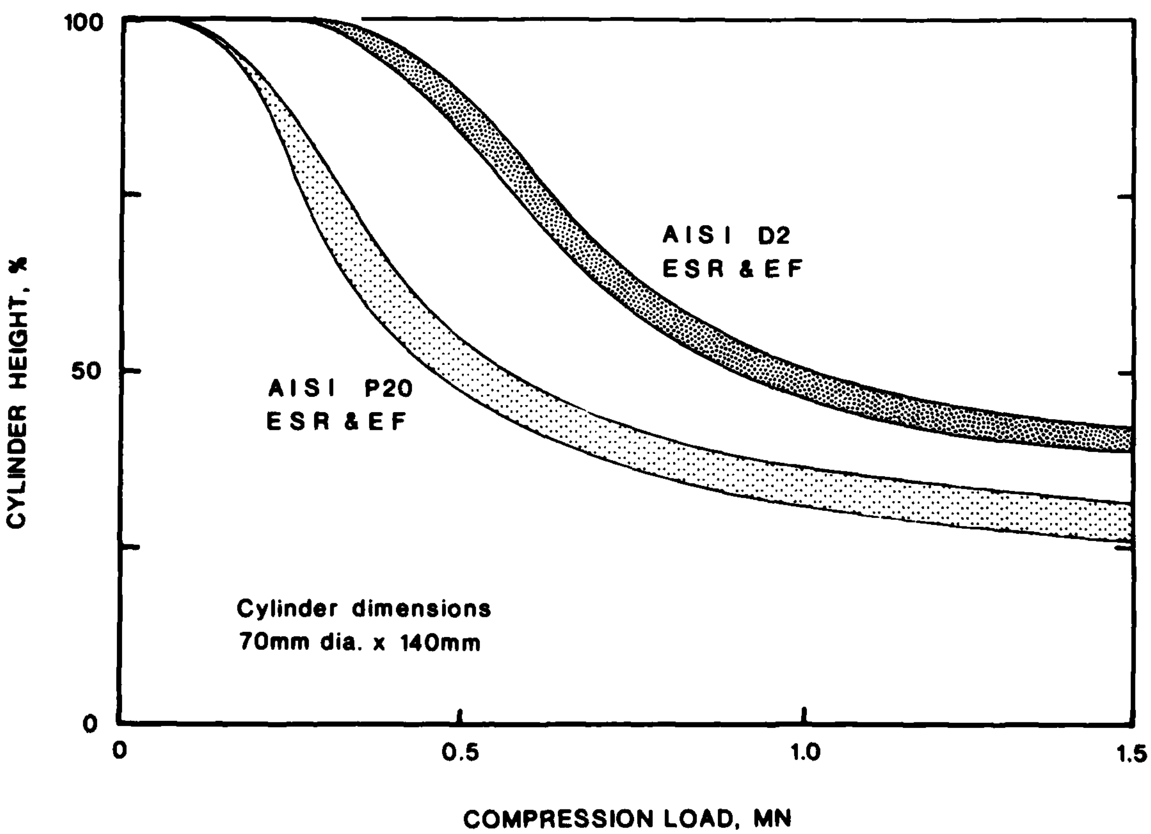


FIG. 2 Typical hot working curves for two alloy steels showing that the process route had little influence on hot workability and the superior hot workability of the lower alloy AISI P20 steel.

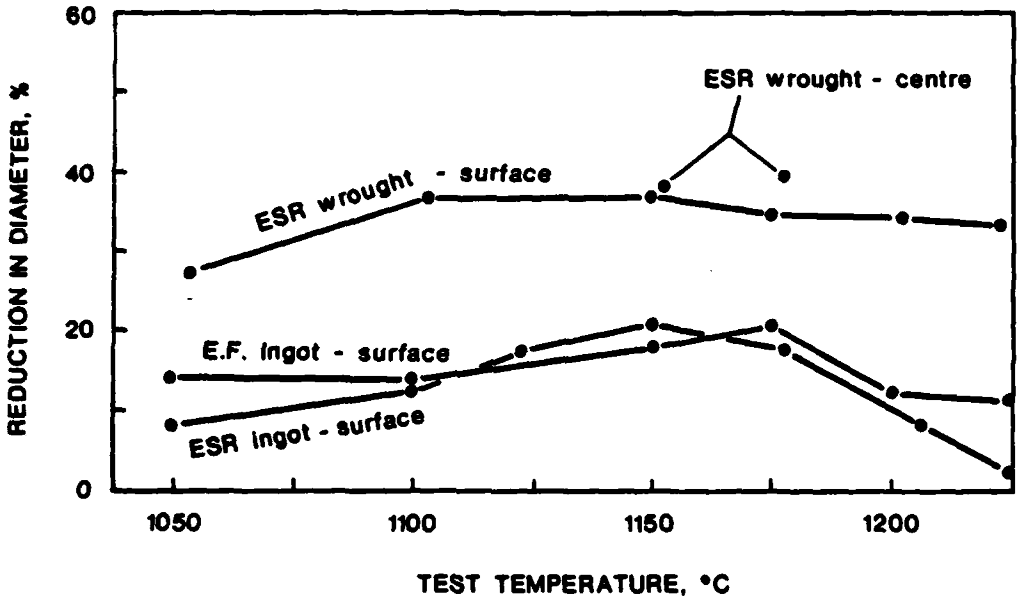
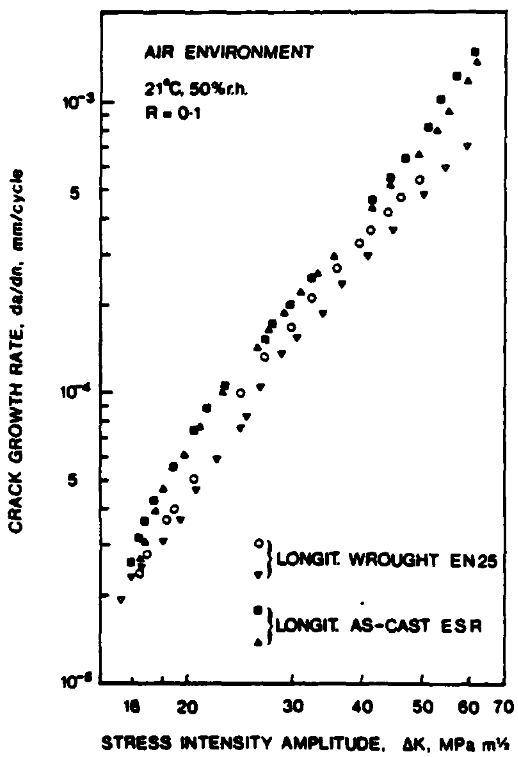
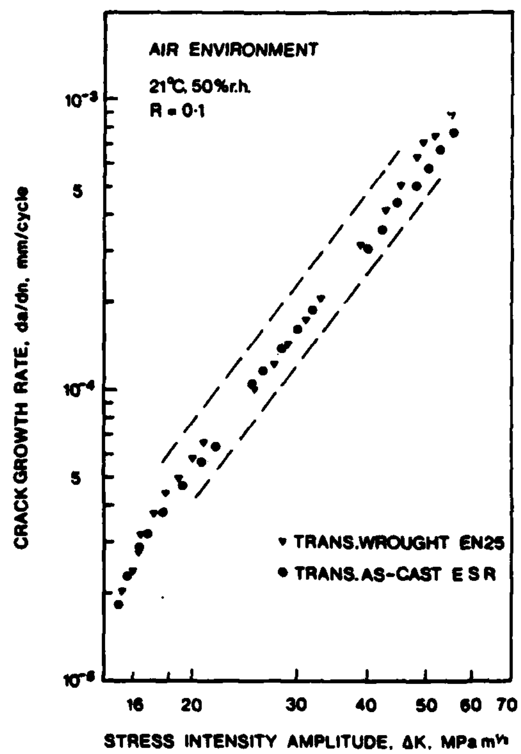


FIG. 3 Hot impact tensile test results of AISI D2 steel.



(a)

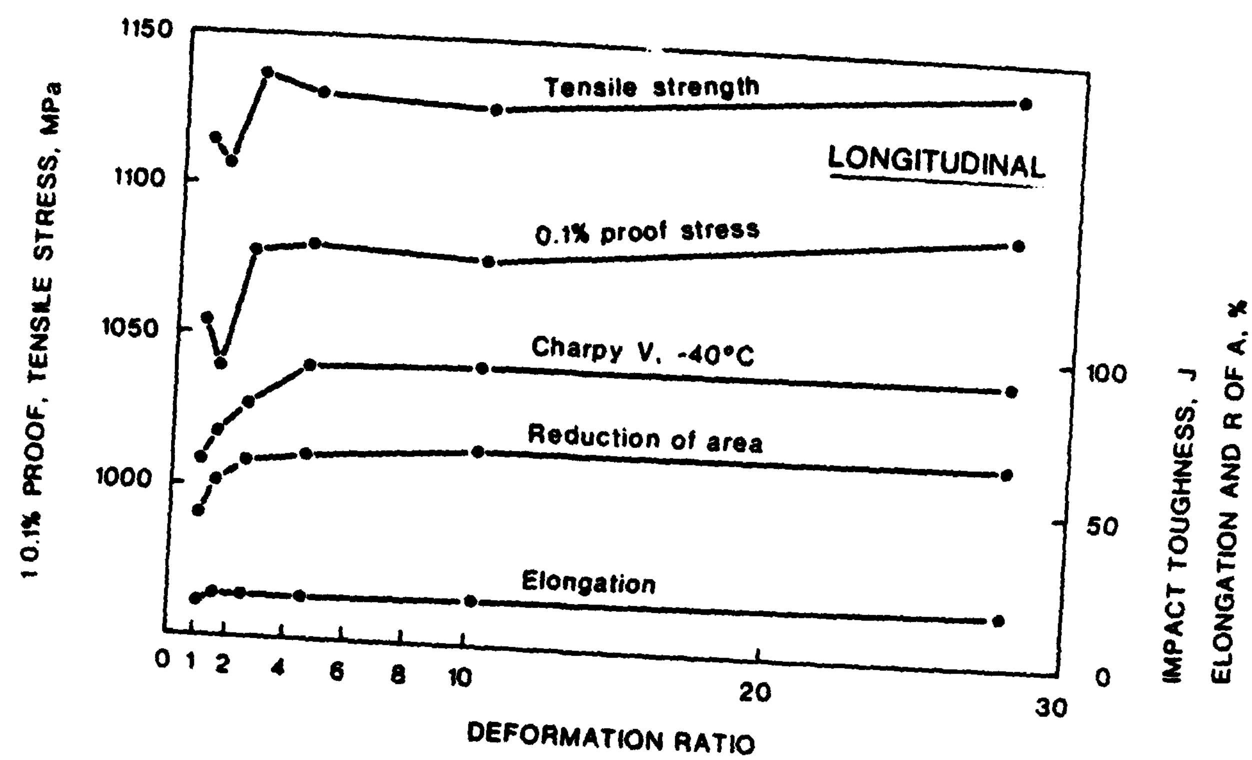


(b)

FIG. 4 Fatigue crack growth characteristics of En25 steel, comparing as-cast ESR material with wrought electric furnace material having a similar sulphur content.

(a) Crack growth in the longitudinal direction.

(b) Transverse growth, with the spread of longitudinal data indicated by broken lines.



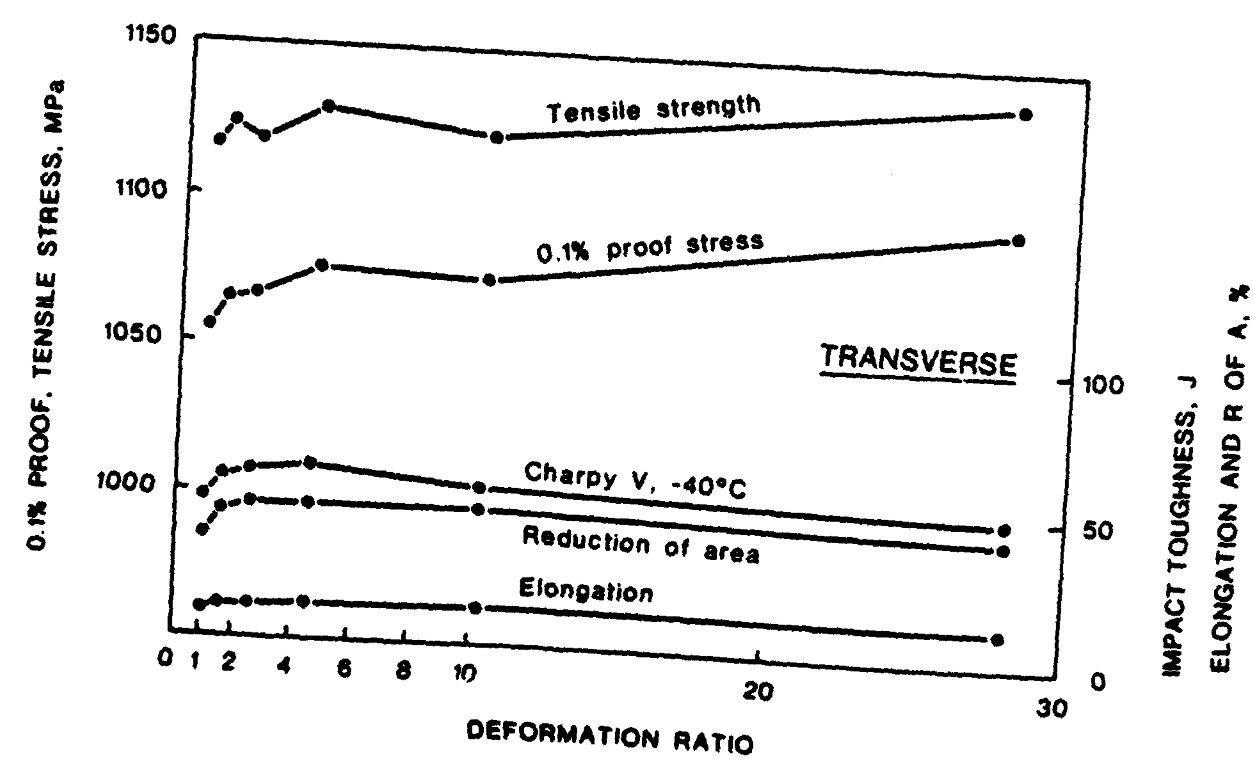


FIG. 5 Influence of deformation ratio on the longitudinal and transverse mechanical properties of ESR En25 steel. Original ingot diameter 800 mm.

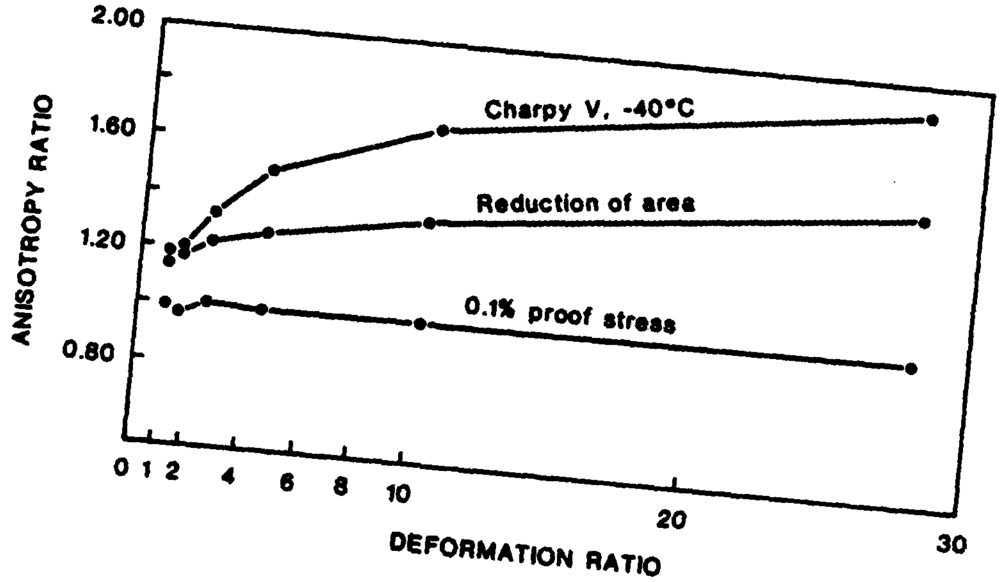


FIG. 6 Influence of deformation ratio on the longitudinal/transverse anisotropy ratio.

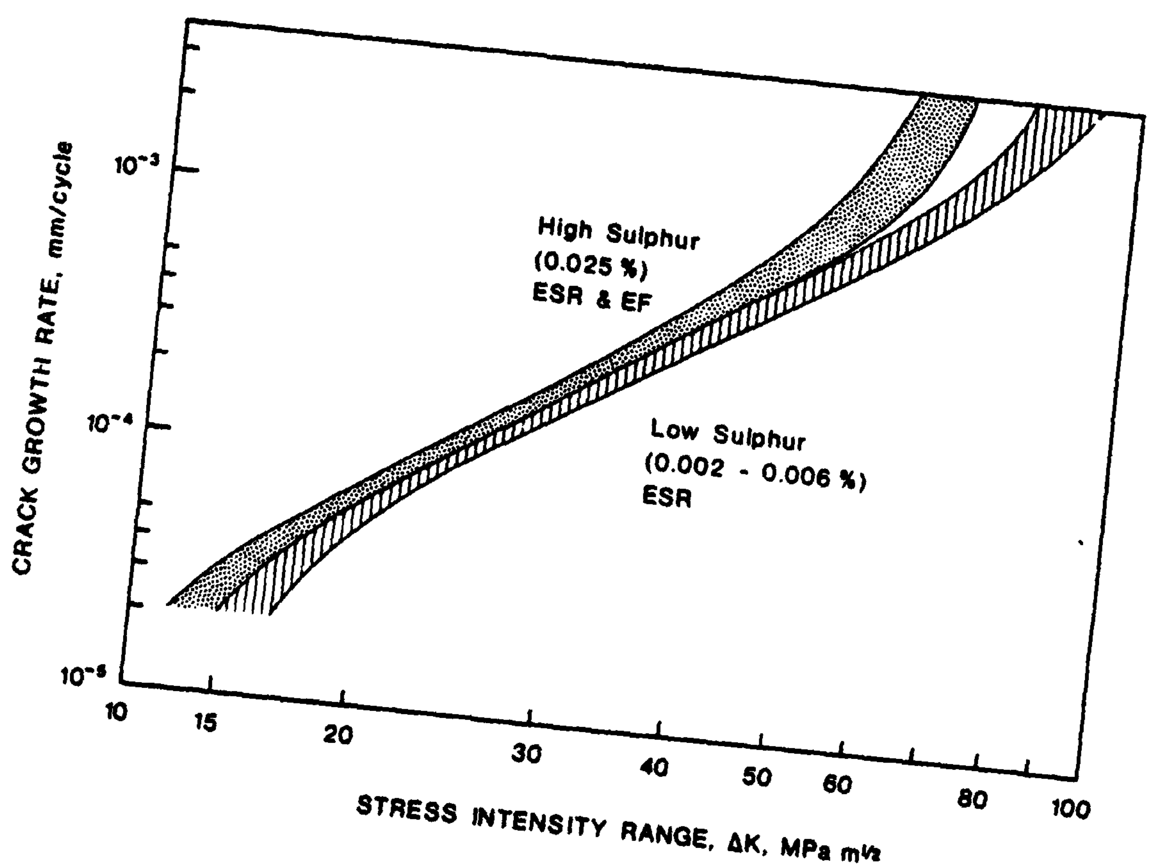


FIG. 7 Influence of sulphur content on fatigue crack growth rates in gun steel.

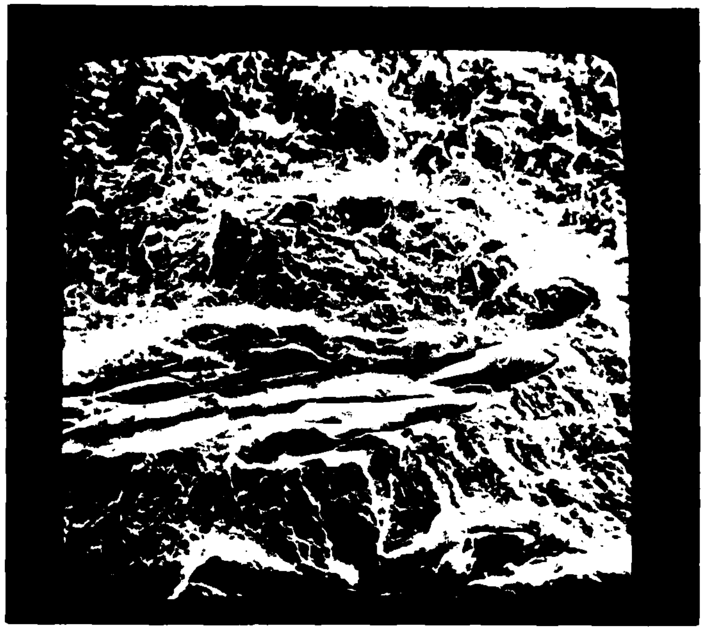


FIG. 8 Fatigue fracture surface of a high-sulphur (0.025% S) steel, showing a pocket of large MnS inclusions common in this sample. Voids are visible around these particles (Electric furnace steel).

SEM X400

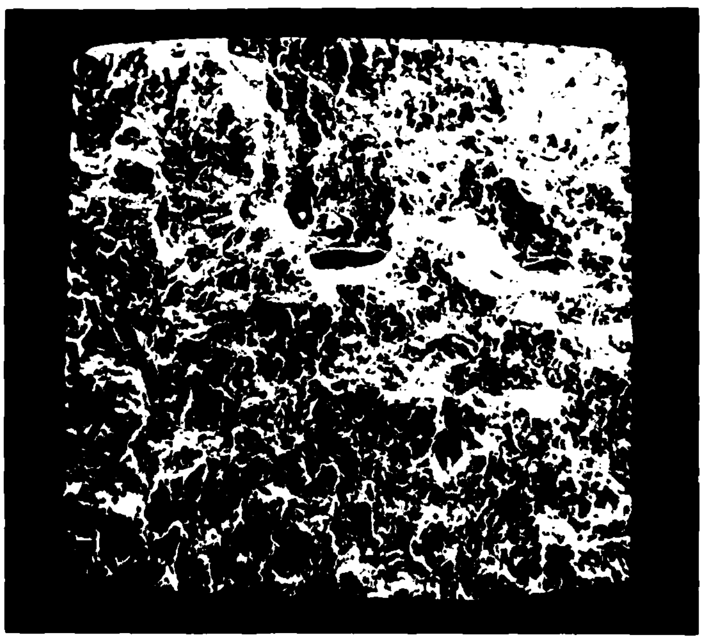


FIG. 9 Fatigue fracture surface of a low-sulphur (0.006% S) steel, showing the largest inclusions found in this sample (ESR steel).

SEM X400

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